



Naval Fuels & Lubricants

Cross Functional Team

Qualification Report

Joint Oil Analysis Program Spectrometer Standards VHG Labs Inc. Qualification Report For D19-0, D3-100 and D12-XXX Series Standards

NF&LCFT REPORT 441/13-010

12 September 2013

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NAVAIR Public Release 2013-843

Distribution Statement A - Approved for public release; distribution is unlimited

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EXECUTIVE SUMMARY

The Joint Oil Analysis Program recently revised the specification for their spectrometric oil standards in order to incorporate an ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy) test method and to transition to commercially manufactured spectrometric oil standards. Previously a Rotrode-AES (Atomic Emission Spectrometry) test method was the only elemental test method used to verify the quality of the spectrometric oil standards. The Rotrode-AES test method was a labor and time intensive process that did not exhibit a high level of accuracy and repeatability. The JOAP wanted to incorporate an ICP-AES test method in order to obtain a more accurate determination of the true concentration of the elements in the spectrometric oil standards and to reduce the cost and time needed to conduct quality assurance. Both the Rotrode-AES and ICP-AES test methods are now part of the qualification inspection in order to ensure that commercially manufactured spectrometric oil standards are identical to those that have been produced in-house by the JOAP.

This report covers the qualification testing of Type D19-0, Type D3-100, and Type D12-XXX candidate spectrometer standards submitted by VHG Labs Inc. against the requirements of MIL-DTL-85694A. In general the candidate spectrometer standards met the requirements of the specification. A few specific data points were analyzed and waived based on alternative testing and analysis. As a result of this testing and analysis effort the VHG Lab Inc. Type D19-0, Type D3-100, and Type D12-XXX series spectrometer standards have been qualified to the specification.

Since this qualification testing effort was the first time the full range of data required by MIL-DTL-85694A has been collected, analysis of the data has lead to the conclusion that several of the requirements of the specification are incorrect and must be revised. Specific recommended changes to the specification will be included in the follow-on report covering the qualification testing of the D19-XXX series candidates submitted by VHG Labs Inc.

LIST OF ACRONYMS/ABBREVIATIONS

Joint Oil Analysis Program.....	JOAP
Joint Oil Analysis Program-Coordinating Group	JOAP-CG
Rotrode Atomic Emission Spectroscopy	Rotrode-AES
Inductively Coupled Plasma Atomic Emission Spectroscopy.....	ICP-AES

Joint Oil Analysis Program Spectrometer Standards VHG Labs Inc. Qualification Report For D19-0, D3-100 and D12-XXX Series Standards

1.0 BACKGROUND

The Joint Oil Analysis Program recently revised the specification for their spectrometric oil standards in order to incorporate an ICP-AES test method and to transition to commercially manufactured spectrometric oil standards. Previously a Rotrode-AES test method was the only elemental test method used to verify the quality of the spectrometric oil standards. The Rotrode-AES test method was a labor and time intensive process that did not exhibit a high level of accuracy and repeatability. The JOAP wanted to incorporate an ICP-AES test method in order to obtain a more accurate determination of the true concentration of the elements in the spectrometric oil standards and to reduce the cost and time needed to conduct quality assurance. Both the Rotrode-AES and ICP-AES test methods are now part of the qualification inspection in order to ensure that commercially manufactured spectrometric oil standards are identical to those that have been produced in-house by the JOAP.

VHG Labs Inc. submitted Type D19-0, Type D12-XXX series, and Type D3-100 spectrometric oil standards that were manufactured to meet the requirements of MIL-DTL-85694A. Testing was accomplished at the NAVAIR Patuxent River MD and Pensacola FL test facilities in accordance with the latest revision of MIL-DTL-85694A.

2.0 OBJECTIVE

The objective of this testing is to qualify a commercial vendor, VHG Labs Inc, for the production of blended spectrometric oil standards that are used in calibrating and verifying the calibration of spectrometers used in spectrometric analysis of metallic elements found in oils and other fluids.

3.0 APPROACH

The test requirements and methods for qualification inspection are specified in Tables I through III.

TABLE I. Physical Property Requirements and Test Methods.

Physical property	Requirement	Test Method
Density at 60 °F	report	ASTM D4052
Viscosity (mm ² /s at 100 °C)	16.3 min., 21.9 max.	ASTM D445
Viscosity Index, Min	85	ASTM D2270
Trace Sediment	0.005 ml/100 mL Oil max.	ASTM D2273
Pour point	-12 °C max.	ASTM D97
Flash point, minimum (COC)	243 °C	ASTM D92

TABLE II. Rotrode-AES (ASTM D6595) Accuracy and Repeatability Requirements

Element	Accuracy Index (AI)												Repeatability Index (RI)											
	0	5	10	30	50	80	100	120	300	500	700	900	0	5	10	30	50	80	100	120	300	500	700	900
Al	0.6	0.9	2.1	3.3	4.5	5.7	7.5	9.3	33.0	45.0	57.0	69.0	0.5	0.6	0.7	1.5	2.5	3.5	5.0	8.0	15.0	25.0	35.0	45.0
Cr	0.6	0.9	2.1	3.3	4.5	5.7	7.2	8.7	33.0	45.0	57.0	69.0	0.5	0.6	0.7	1.5	2.5	3.5	5.0	8.0	15.0	25.0	35.0	45.0
Cu	0.8	1.2	2.1	4.5	7.1	9.6	13.7	17.7	45.0	70.0	95.0	120.0	0.5	0.7	1.0	2.7	4.5	6.5	9.0	11.0	27.0	45.0	63.0	81.0
Fe	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.9	2.4	4.0	6.0	8.0	10.0	24.0	40.0	56.0	72.0
Pb	1.2	1.5	2.1	3.3	5.0	6.6	9.2	11.7	33.0	50.0	67.0	84.0	0.9	1.0	1.0	2.0	3.1	4.5	6.0	9.0	18.0	30.0	42.0	54.0
Mg	0.8	1.2	2.1	4.5	7.1	9.6	13.7	17.7	45.0	70.0	95.0	120.0	0.5	1.0	1.6	4.8	8.0	8.5	9.0	11.0	27.0	45.0	63.0	81.0
Ni	0.6	0.9	2.1	3.3	4.5	5.7	7.5	9.3	33.0	45.0	57.0	69.0	0.5	0.6	0.7	1.5	2.5	3.5	5.0	8.0	15.0	25.0	35.0	45.0
Si	0.6	0.9	2.1	3.3	4.5	5.7	7.5	9.3	33.0	45.0	57.0	69.0	0.5	0.6	0.7	1.5	2.5	3.5	5.0	8.0	15.0	25.0	35.0	45.0
Ag	1.0	1.8	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.9	2.4	4.0	6.0	8.0	10.0	24.0	40.0	56.0	72.0
Na	0.8	2.0	3.3	8.7	14.1	19.5	28.5	37.5	87.0	140.0	193.0	246.0	0.5	1.0	1.6	4.8	8.0	12.0	16.0	20.0	48.0	80.0	112.0	144.0
Sn	1.2	1.5	2.1	3.3	5.0	6.6	9.2	11.7	33.0	50.0	67.0	84.0	0.5	1.0	1.0	2.0	3.1	4.5	6.0	9.0	18.0	30.0	42.0	54.0
Ti	0.6	0.9	2.1	3.3	4.7	6.0	8.7	11.4	33.0	47.0	61.0	75.0	0.5	0.6	0.7	1.8	3.0	4.3	6.0	9.0	18.0	30.0	42.0	54.0
B	0.6	0.9	2.1	3.3	4.7	6.0	8.7	11.4	33.0	47.0	61.0	75.0	0.5	0.6	0.7	1.8	3.0	4.3	6.0	9.0	18.0	30.0	42.0	54.0
Mo	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.9	2.4	4.0	5.5	8.0	10.0	24.0	40.0	56.0	72.0
Zn	0.8	1.5	2.4	6.2	9.9	13.7	19.5	25.4	62.0	99.0	136.0	173.0	0.5	0.8	1.3	3.6	6.0	9.0	12.0	18.0	36.0	60.0	84.0	108.0
Ba	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.7	1.8	3.0	4.5	6.0	9.0	18.0	30.0	42.0	54.0
Cd	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.7	1.8	3.0	4.5	6.0	9.0	18.0	30.0	42.0	54.0
Mn	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.7	1.8	3.0	4.5	6.0	9.0	18.0	30.0	42.0	54.0
V	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.8	0.6	0.9	1.8	3.0	4.5	6.0	9.0	18.0	30.0	42.0	54.0
Ca	0.5	1.2	1.5	3.3	5.5	8.3	10.0	12.6	32.0	53.0	73.9	95.0	0.5	0.6	0.8	1.8	2.8	3.5	3.8	4.1	10.0	20.0	29.8	40.0
P	4.4	6.0	7.5	15.5	18.2	19.4	20.0	21.6	35.0	60.0	82.3	105.0	1.1	1.5	1.9	5.5	7.5	9.3	10.0	12.5	15.0	25.0	32.6	40.0

Note: The column headings, 0, 5, 10, 30, 50, 80, 100, 120, 300, 500, 700 and 900 denote the nominal concentration in parts per million (ppm).

TABLE III. ICP-AES (ASTM D5185) Accuracy Requirements

	Type D19-0	Type D19-XXX and D21-XXX											Type D12-XXX						Type D3-100
Element	0	5	10	30	50	80	100	120	300	500	700	900	5	10	30	50	100	300	100
Al	1.0	1.4	1.8	2.9	3.5	4.3	4.7	5.1	7.4	9.1	10.4	11.5	1.4	1.8	2.9	3.5	4.7	7.4	-----
Cr	1.0	0.6	1.0	2.2	3.2	4.5	5.4	6.2	12.3	18.0	23.1	27.9	0.6	1.0	2.2	3.2	5.4	12.3	-----
Cu	1.0	0.5	1.0	2.7	4.2	6.5	7.9	9.4	21.5	34.3	46.6	58.6	0.5	1.0	2.7	4.2	7.9	21.5	-----
Fe	1.0	0.5	0.8	2.0	3.0	4.3	5.2	6.0	12.5	18.8	24.5	30.0	0.5	0.8	2.0	3.0	5.2	12.5	-----
Pb	1.0	2.7	3.3	4.8	5.6	6.5	7.0	7.4	9.9	11.7	13.0	14.1	2.7	3.3	4.8	5.6	7.0	9.9	-----
Mg	1.0	0.6	1.2	3.0	4.6	6.9	8.4	9.8	21.6	33.5	44.8	55.6	0.6	1.2	3.0	4.6	8.4	21.6	-----
Ni	1.0	1.1	1.6	2.8	3.5	4.5	5.0	5.4	8.5	10.9	12.9	14.6	1.1	1.6	2.8	3.5	5.0	8.5	-----
Si	1.0	2.0	2.4	3.1	3.6	4.1	4.3	4.5	5.7	6.5	7.1	7.6	2.0	2.4	3.1	3.6	4.3	5.7	-----
Ag	1.0	0.6	1.0	2.5	3.9	5.7	6.9	8.0	17.1	26.1	34.5	42.5	0.6	1.0	2.5	3.9	6.9	17.1	-----
Na	1.0	1.4	2.2	4.6	6.5	8.8	10.2	11.5	21.1	29.6	37.0	43.7	1.4	2.2	4.6	6.5	10.2	21.1	-----
Sn	1.0	3.2	3.5	4.3	4.7	5.1	5.3	5.4	6.3	6.9	7.3	7.6	3.2	3.5	4.3	4.7	5.3	6.3	-----
Ti	1.0	1.0	1.3	1.9	2.3	2.7	3.0	3.2	4.5	5.4	6.1	6.7	0.9 ^A	1.2 ^A	1.8 ^A	2.2 ^A	2.9 ^A	4.3 ^A	-----
B	1.0	1.3	2.6	7.8	13.0	20.8	26.0	31.2	78.0	130.0	182.0	234.0	-----	-----	-----	-----	-----	-----	29.9 ^B
Mo	1.0	0.9	1.5	3.1	4.5	6.2	7.3	8.3	15.7	22.5	28.4	33.9	-----	-----	-----	-----	-----	-----	6.5 ^C
Zn	1.0	0.6	1.1	3.0	4.7	7.1	8.6	10.1	22.7	35.6	47.8	59.7	-----	-----	-----	-----	-----	-----	8.6
Ba	1.0	0.7	1.1	2.3	3.2	4.3	5.0	5.7	10.4	14.5	18.1	21.4	-----	-----	-----	-----	-----	-----	-----
Cd	1.0	0.8	1.5	4.5	7.5	12.0	15.0	18.0	45.0	75.0	105.0	135.0	-----	-----	-----	-----	-----	-----	-----
Mn	1.0	0.5	1.0	3.0	5.0	3.0	10.0	5.0	30.0	50.0	50.0	90.0	-----	-----	-----	-----	-----	-----	-----
V	1.0	0.3	0.6	1.8	3.1	4.9	6.1	7.3	18.3	30.5	42.7	54.9	-----	-----	-----	-----	-----	-----	-----
Ca	1.0	0.5	1.0	3.0	5.0	8.0	10.0	12.0	30.0	50.0	19.2	90.0	-----	-----	-----	-----	-----	-----	-----
P	1.0	3.3	4.9	9.3	12.6	16.5	18.8	20.9	35.5	47.8	58.1	67.2	-----	-----	-----	-----	-----	-----	-----

Note: The column headings, 0, 5, 10, 30, 50, 80, 100, 120, 300, 500, 700, and 900 denote the nominal concentration in parts per million (ppm).

^A - Ti concentration = 0.92X, where X = nominal concentration of spectrometric oil standard in ppm.

^B - B concentration = 1.15X, where X = nominal concentration of spectrometric oil standard in ppm.

^C - Mo concentration = 0.85X, where X = nominal concentration of spectrometric oil standard in ppm.

The density, viscosity, viscosity index, trace sediment, pour point, and flash point test methods were conducted in accordance with the corresponding ASTM test method.

The Rotrode-AES test method was conducted in accordance with ASTM D6595. Testing of candidate spectrometric oil standards was done as a statistical analysis and performed on the data generated by the spectrometric analysis to provide an accuracy index and repeatability index for each element involved. The results were obtained with a JOAP-CG approved Spectroil M/N spectrometer (S/N 0620). The testing consisted of twelve replicates of each candidate spectrometric standard.

The ICP-AES test method was conducted in accordance with ASTM D5185. Testing of spectrometric oil standards was done as a statistical analysis and performed on the data generated by the spectrometric analysis to provide an accuracy index for each element involved. The results were obtained with a JOAP-CG approved ICP spectrometer (Perkin Elmer 4300DV). The ICP spectrometer was calibrated from 0 ppm to 10 ppm using JOAP D19 reference spectrometric standards. Candidate spectrometric oil standards were diluted on a weight-by-weight basis with a suitable ICP solvent. Candidate spectrometric oil standards in concentrations 5 ppm, 10 ppm, 30 ppm, and 50 ppm were diluted tenfold while candidate standards in concentrations 80 ppm, 100 ppm, 120 ppm, 300 ppm, 500 ppm, 700 ppm, and 900 ppm were diluted to 5 ppm. The testing consisted of five replicates of each candidate spectrometric standard.

4.0 DISCUSSION

This section details the analysis of the data according to the requirements published in the specification, MIL-DTL-85694A. An explanation of instrumental limitation and anomalies are provided in the event that the requirements are not met. Specification requirements that are not met will be subject to revision due to factors that influenced some chemical and physical properties that were not taken into consideration during the development of the specification.

4.1 Type D19-0

The Type D19-0 spectrometric oil standard submitted by the candidate met the requirements for density at 60°F (ASTM D4052), viscosity at 100°C (ASTM D445), viscosity index (ASTM 2270), trace sediment (ASTM D2273), pour point (ASTM D97), and flash point (ASTM D92).

TABLE IV. Candidate Type D19-0 Physical Property Results

TEST	METHOD	UNITS	SAMPLE
			Type D19-0
			D19-0
Density at 60°F	ASTM D4052	kg/L	0.8835
Viscosity at 100°C	ASTM D445	cSt	19.1
Viscosity @ 40°C	ASTM D445	cSt	219.7
Viscosity Index	ASTM D2270	No Units	98
Water & Sediment	ASTM D2273	%	0
Pour Point	ASTM D97	°C	-15
Flash Point	ASTM D92	°C	261

The Type D19-0 spectrometric oil standard submitted by the candidate did not meet the requirement for Rotrode-AES (ASTM D6595) and ICP-AES (ASTM D5185). Elevated barium levels were observed in the Rotrode-AES data and not in the ICP-AES data. This suggests the elevated Rotrode-AES reading (barium) is an instrumental anomaly and the failure can be waived. Elevated sodium and boron levels were observed in the ICP-AES data and not in the Rotrode-AES data. This suggests the elevated ICP-AES readings (sodium and boron) are instrumental anomalies and the failures can be waived.

TABLE V. Candidate Type D19-0 Rotrode-AES Results

TEST	METHOD	UNITS	SAMPLE	
			Type D19-0	
			D19-0	
Trace Metal:	ASTM D6595	ppm	Mean	Stdev
Al			0.0	0.1
Cr			0.1	0.1
Cu			0.0	0.0
Fe			0.0	0.0
Pb			0.2	0.1
Mg			0.0	0.0
Ni			0.1	0.1
Si			0.2	0.4
Ag			0.0	0.0
Na			0.0	0.0
Sn			0.1	0.2
Ti			0.0	0.0
B			0.0	0.0
Mo			0.2	0.2
Zn			0.0	0.0
Ba			1.0	0.6
Cd			0.1	0.0
Mn			0.1	0.1
V			0.2	0.3

NOTE: Red numbers indicate the requirement not being met

TABLE VI. Candidate Type D19-0 ICP-AES Results

TEST	METHOD	UNITS	SAMPLE
			Type D19-0
			D19-0
ICP	ASTM D5185	ppm	Mean
Al			0.1
Cr			-0.2
Cu			0.2
Fe			0.3
Pb			0.8
Mg			0.2
Ni			0.7
Si			0.3
Ag			0.0
Na			1.7
Sn			0.1
Ti			0.5
B			1.4
Mo			-0.4
Zn			0.1
Ba			-0.4
Cd			-0.4
Mn			0.4
V			0.5

NOTE: Red numbers indicate the requirement not being met

The Type D19-0 spectrometric oil standard submitted by the candidate satisfactorily passed the qualification inspection.

4.2 Type D12-XXX Series

All of the Type D12-XXX spectrometric oil standards submitted by the candidate met the requirements for density at 60°F (ASTM D4052), viscosity at 100°C (ASTM D445), viscosity index (ASTM 2270), trace sediment (ASTM D2273), and pour point (ASTM D97).

TABLE VII. Candidate Type D12-XXX Physical Property Results

TEST	METHOD	UNITS	SAMPLES					
			Type D12-XXX					
			D12-5	D12-10	D12-30	D12-50	D12-100	D12-300
Density at 60°F	ASTM D4052	kg/L	0.8825	0.8838	0.8846	0.8847	0.8844	0.8887
Viscosity at 100°C	ASTM D445	cSt	19.1	19.1	19.0	19.0	18.9	18.4
Viscosity @ 40°C	ASTM D445	cSt	220.4	220.1	219.3	218.5	216.6	208.5
Viscosity Index	ASTM D2270	No Units	97	98	97	98	98	97
Water & Sediment	ASTM D2273	%	0	0	0	0	0	0
Pour Point	ASTM D97	°C	-18	-18	-12	-12	-15	-15

All of the Type D12-XXX spectrometric oil standards with the exception of D12-300 met the requirement for flash point (ASTM D92). The high concentration of the metallo-organic concentrates in D12-300 has an effect on the flash point which resulted in the requirement for flash point not being met. The significance of the effect was not taken into consideration during the development of the original specification requirements therefore the flash point requirement for D12-300 will require revision. At this time we are waiving the flash point requirement for D12-300.

TABLE VIII. Candidate Type D12-XXX Flash Point Results

TEST	METHOD	UNITS	SAMPLES					
			Type D12-XXX					
			D12-5	D12-10	D12-30	D12-50	D12-100	D12-300
Flash Point	ASTM D92	°C	255	255	259	263	249	221

NOTE: Red numbers indicate the requirement not being met

All of the Type D12-XXX spectrometric oil standards with the exceptions of D12-100 and D12-300 met the requirement for Rotrode-AES (ASTM D6595). The Rotrode-AES chromium accuracy for D12-100 and D12-300 were identified as being outside of the Rotrode-AES requirement. Following standard JOAP quality assurance procedure, the D12-100 and D12-300 were diluted to 50 ppm using D19-0 and retested to address the chromium accuracy and titanium repeatability. Upon retesting, the Rotrode-AES requirements for chromium accuracy and titanium repeatability were met.

TABLE IX. Candidate Type D12-XXX Rotrode-AES Results

TEST	METHOD	UNITS	SAMPLES											
			Type D12-XXX											
			D12-5		D12-10		D12-30		D12-50		D12-100		D12-300	
Trace Metal:	ASTM D6595	ppm	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev
Al			5.0	0.3	9.9	0.4	32.0	0.8	50.0	1.6	104.0	3.5	324.7	10.9
Cr			5.5	0.3	11.0	0.7	32.0	1.5	52.0	2.3	111.0	3.3	347.8	11.5
Cu			5.2	0.1	10.0	0.4	32.0	0.8	55.0	1.6	104.0	6.1	315.1	20.7
Fe			5.1	0.3	10.0	0.3	31.0	1.4	52.0	2.4	107.0	5.9	322.7	15.5
Pb			5.1	0.2	10.0	0.2	31.0	0.8	53.0	1.7	105.0	4.0	317.1	11.5
Mg			5.1	0.3	10.0	0.4	30.0	1.9	52.0	3.1	103.0	6.8	313.8	10.1
Ni			5.7	0.1	11.0	0.4	31.0	0.6	52.0	1.2	106.0	3.2	328.2	14.1
Si			5.9	0.3	12.0	0.7	33.0	1.4	53.0	2.5	106.0	4.7	318.7	11.7
Ag			5.0	0.1	9.9	0.3	33.0	0.9	54.0	2.3	104.0	7.2	290.2	15.5
Na			5.6	0.4	10.0	0.9	34.0	2.5	56.0	4.4	104.0	5.4	335.1	16.4
Sn			4.9	0.7	10.0	0.5	31.0	1.2	51.0	1.5	101.0	2.4	308.5	7.6
Ti			4.9	0.3	9.8	0.3	30.0	1.7	52.0	2.6	103.0	5.7	306.8	20.4

NOTE: Red numbers indicate the requirement not being met

TABLE X. Retest of Candidate Type D12-XXX Rotrode-AES Results

TEST			SAMPLES											
			Type D12-XXX											
			D12-5		D12-10		D12-30		D12-50		D12-100		D12-300	
Trace Metal:	ASTM D6595	ppm	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev
Al											50.3	1.4	49.8	1.4
Cr											53.0	2.0	53.0	2.3
Cu											54.4	1.0	53.4	1.9
Fe											55.2	1.4	54.5	2.9
Pb											54.6	1.2	54.2	1.7
Mg											54.3	2.2	53.6	3.7
Ni											53.9	1.0	53.8	1.2
Si											57.1	1.3	56.4	2.4
Ag											53.5	1.8	51.8	2.5
Na											54.5	2.0	53.4	2.9
Sn											52.6	1.1	52.4	1.5
Ti											53.5	2.2	52.5	2.9

NOTE: Red numbers indicate the requirement not being met

The D12-10 and D12-30 spectrometric oil standards met the requirement for ICP-AES (ASTM D5185) while the D12-5, D12-50, D12-100, and D12-300 did not. Elevated sodium levels were observed in the ICP-AES D12-5 data and not in the Rotrode-AES D12-5 data. This suggests the elevated ICP-AES reading (sodium) is an instrumental anomaly and the failure can be waived. Upon further analysis, it was determined that the accuracy listed in the specification did not accurately take into consideration the dilution factors and resulted in the D12-50, D12-100, and D12-300 spectrometric oil standards not meeting the ICP-AES requirements. Therefore the dilution factors were removed from each sample and the ICP-AES data were reevaluated using the appropriate limits for the given concentration. Table XII lists for each element in the Type D12-XXX spectrometric oil standards the average ICP result without the dilution factor, the expected concentration, and the appropriate limit as originally published in the specification.

TABLE XI. Candidate Type D12-XXX ICP-AES Results

TEST			SAMPLES					
			Type D12-XXX					
			D12-5	D12-10	D12-30	D12-50	D12-100	D12-300
ICP	ASTM D5185	ppm	Mean	Mean	Mean	Mean	Mean	Mean
Al			4.8	9.6	28.3	51.7	102.7	319.9
Cr			4.5	9.2	27.8	52.8	104.9	296.4
Cu			4.9	9.6	28.0	52.4	102.9	285.1
Fe			4.9	9.6	28.0	52.6	103.7	294.7
Pb			4.9	9.9	28.7	54.1	105.6	327.1
Mg			4.9	9.7	28.7	52.5	104.6	302.2
Ni			5.5	10.7	30.1	54.2	108.4	335.5
Si			5.4	10.1	28.8	52.9	104.2	312.6
Ag			5.2	10.2	29.4	53.7	108.4	327.1
Na			7.4	12.1	31.3	53.9	106.3	353.9
Sn			4.8	9.6	28.0	52.7	102.9	301.9
Ti			5.1	9.6	27.3	48.3	97.5	285.7

NOTE: Red numbers indicate the requirement not being met

All of the Type D12-XXX spectrometric oil standards with the exception of D12-5 met the requirement for ICP-AES (ASTM D5185) once the dilution factors were removed. The elevated sodium levels are still viewed as an instrumental anomaly and the failure can be waived.

TABLE XII. Reevaluation of Candidate Type D12-XXX ICP-AES Results

TEST	METHOD	UNITS	SAMPLES					
			D12-5	D12-10	D12-30	D12-50	D12-100	D12-300
ICP	ASTM D5185	ppm	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit
Al			0.5 / 0.5 / 0.1	0.9 / 1 / 0.3	2.7 / 2.9 / 0.8	5.6 / 5.1 / 1.4	5.1 / 5 / 1.4	5.3 / 5 / 1.4
Cr			0.5 / 0.5 / 0.1	0.9 / 1 / 0.1	2.7 / 2.9 / 0.3	5.4 / 5.1 / 0.6	5.2 / 5 / 0.6	5.3 / 5 / 0.6
Cu			0.5 / 0.5 / 0.1	0.9 / 1 / 0.1	2.7 / 2.9 / 0.3	5.2 / 5.1 / 0.5	5.1 / 5 / 0.5	5.2 / 5 / 0.5
Fe			0.5 / 0.5 / 0.1	0.9 / 1 / 0.1	2.7 / 2.9 / 0.3	5.3 / 5.1 / 0.5	5.1 / 5 / 0.5	5.3 / 5 / 0.5
Pb			0.5 / 0.5 / 0.3	1 / 1 / 0.5	2.7 / 2.9 / 1.5	5.7 / 5.1 / 2.7	5.2 / 5 / 2.7	5.4 / 5 / 2.7
Mg			0.5 / 0.5 / 0.1	0.9 / 1 / 0.1	2.7 / 2.9 / 0.3	5.3 / 5.1 / 0.6	5.2 / 5 / 0.6	5.3 / 5 / 0.6
Ni			0.5 / 0.5 / 0.1	1 / 1 / 0.2	2.9 / 2.9 / 0.6	5.9 / 5.1 / 1.1	5.4 / 5 / 1.1	5.5 / 5 / 1.1
Si			0.5 / 0.5 / 0.2	1 / 1 / 0.4	2.7 / 2.9 / 1.1	5.5 / 5.1 / 2	5.2 / 5 / 2	5.3 / 5 / 2
Ag			0.5 / 0.5 / 0.1	1 / 1 / 0.1	2.8 / 2.9 / 0.3	5.6 / 5.1 / 0.6	5.4 / 5 / 0.6	5.4 / 5 / 0.6
Na			0.7 / 0.5 / 0.1	1.2 / 1 / 0.3	3 / 2.9 / 0.8	5.9 / 5.1 / 1.4	5.3 / 5 / 1.4	5.4 / 5 / 1.4
Sn			0.5 / 0.5 / 0.3	0.9 / 1 / 0.6	2.7 / 2.9 / 1.8	5.3 / 5.1 / 3.3	5.1 / 5 / 3.2	5.3 / 5 / 3.2
Ti			0.5 / 0.5 / 0.1	0.9 / 1 / 0.2	2.6 / 2.9 / 0.6	5 / 5.1 / 1	4.8 / 5 / 1	4.9 / 5 / 1

NOTE: Red numbers indicate the requirement not being met

The Type D12-XXX spectrometric oil standards submitted by the candidate satisfactorily passed the qualification inspection.

4.3 Type D3-100

The Type D3-100 spectrometric oil standard submitted by the candidate met the requirements for density at 60°F (ASTM D4052), viscosity at 100°C (ASTM D445), viscosity index (ASTM D2270), trace sediment (ASTM D2273), pour point (ASTM D97), flash point (ASTM D92), Rotrode-AES (ASTM D6595), and ICP-AES (ASTM D5185).

TABLE XIII. Candidate Type D3-100 Chemical and Physical Property Results

TEST	METHOD	UNITS	SAMPLE	
			Type D3-100	
			D3-100	
Density at 60°F	ASTM D4052	kg/L	0.8846	
Viscosity at 100°C	ASTM D445	cSt	18.8	
Viscosity @ 40°C	ASTM D445	cSt	213.9	
Viscosity Index	ASTM D2270	No Units	98	
Water & Sediment	ASTM D2273	%	0	
Pour Point	ASTM D97	°C	-15	
Flash Point	ASTM D92	°C	255	
Trace Metal:	ASTM D6595	ppm	Mean	Stdev
B			98.3	4.4
Mo			102.8	7.9
Zn			91.6	6.2
ICP	ASTM D5185	ppm	Mean	
B			115.0	
Mo			83.6	
Zn			95.8	

The Type D3-100 spectrometric oil standard submitted by the candidate satisfactorily passed the qualification inspection.

5.0 CONCLUSIONS

The following spectrometric oil standards submitted by VHG Labs Inc. have satisfactorily passed the qualification inspection and should be added to the qualified products list for MIL-DTL-85694A:

- Type D19-0
- Type D12-XXX (D12-5, D12-10, D12-30, D12-50, D12-100, D12-300)
- Type D3-100

6.0 RECOMMENDATIONS

A number of issues associated with the specification requirements were brought to light as a result of the qualification testing. Several changes to the specification are therefore under consideration. Detailed recommended changes to MIL-DTL-85694A will be published upon the completion of the qualification testing of Type D19-XXX spectrometric oil standards submitted by VHG Labs Inc. so that all of the information collected during the qualification testing effort can be taken into account.

REPORT DOCUMENTATION PAGE			<i>Form Approved</i> OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.				
1. REPORT DATE (DD-MM-YYYY) 09-12-2013		2. REPORT TYPE Technical		3. DATES COVERED (From - To) 08-20-2011 to 09-12-2013
4. TITLE AND SUBTITLE Joint Oil Analysis Program Spectrometer Standards; VHG Labs Inc. Qualification Report for D19-0, D3-100, and D12-XXX Series Standards			5a. CONTRACT NUMBER N/A	
			5b. GRANT NUMBER N/A	
			5c. PROGRAM ELEMENT NUMBER N/A	
6. AUTHOR(S) Peretich, Michael; Author Eldridge, Geoffrey.; Editor Krizovensky, John ; Editor Mearns, Douglas ; Editor			5d. PROJECT NUMBER N/A	
			5e. TASK NUMBER N/A	
			5f. WORK UNIT NUMBER N/A	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Naval Fuels & Lubricants Cross Functional Team 22229 Elmer Road Patuxent River, MD 20670			8. PERFORMING ORGANIZATION REPORT NUMBER NF&LCFT Report 441/13-010	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) Naval Air Systems Command NPRE Program 22347 Cedar Point Road Patuxent River MD 20670			10. SPONSOR/MONITOR'S ACRONYM(S) N/A	
			11. SPONSOR/MONITOR'S REPORT NUMBER(S) N/A	
12. DISTRIBUTION / AVAILABILITY STATEMENT A Approved for public release; distribution is unlimited.				
13. SUPPLEMENTARY NOTES N/A				
14. ABSTRACT The Joint Oil Analysis Program recently revised the specification for their spectrometric oil standards in order to incorporate an ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy) test method and to transition to commercially manufactured spectrometric oil standards. Previously a Rotrode-AES (Atomic Emission Spectrometry) test method was the only elemental test method used to verify the quality of the spectrometric oil standards. The Rotrode-AES test method was a labor and time intensive process that did not exhibit a high level of accuracy and repeatability. The JOAP wanted to incorporate an ICP-AES test method in order to obtain a more accurate determination of the true concentration of the elements in the spectrometric oil standards and to reduce the cost and time needed to conduct quality assurance. Both the Rotrode-AES and ICP-AES test methods are now part of the qualification inspection in order to ensure that commercially manufactured spectrometric oil standards are identical to those that have been produced in-house by the JOAP. As a result this testing effort the VHG Lab Inc. D19-0, Type D3-100, and Type D12-XXX series spectrometer standards are qualified to the MIL-DTL-85694A specification. Since this qualification testing effort was the first time the full range of data required by MIL-DTL-85694A has been generated, analysis of the data has lead to the conclusion that several of the requirements of the specification are incorrect and must be revised. Specific recommended changes to the specification will be included in the follow-on report covering the qualification testing of the D19-				
15. SUBJECT TERMS Joint Oil Analysis Program, Spectrometer Standards				
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES
a. REPORT UNCLASSIFIED	b. ABSTRACT UNCLASSIFIED	c. THIS PAGE UNCLASSIFIED	Unclassified Unlimited	17
				19a. NAME OF RESPONSIBLE PERSON Douglas F. Mearns
				19b. TELEPHONE NUMBER (include area code) 301-757-3421